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1. Introduction

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This report covers [] the following topics:

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- a. Design of a Urea Plant
- b. Design of an Experimental Laboratory for Severo-Donetsk
- c. Report on Acetic Acid and Esterification
- d. Construction and Installation Plans for Dismantled Leuna Equipment
- e. Questionnaire on Ammonium Nitrate
- f. Memorandum on Kaurit Leim or K-Glue
- g. Report on Silica-Alumina Catalysts
- h. Production of Formaldehyde
- i. Production of Propionaldehyde
- j. Production of Trimethylol Ethane
- k. Production of Glycerine
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- o. Production of Adipic Acid
- p. Report on Oxo Process
- q. Report on Synol Process
- r. Design of Tunnel-Type Drying Oven
- s. Questionnaire on Brown Oxide Catalyst
- t. Production of Ammonia Catalyst
- u. Memorandum on Hydrogen Purification with Alkaline Copper Solution
- v. Production of Higher Alcohols by Aldehyde Reduction

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ARMY & NAVY REVIEWS COMPLETED

25 YEAR RE-REVIEW

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- W. Production of Methanol and Isobutyl Oil
- X. Separation of Alcohols in Isobutyl Oil
- Y. Design of a Pilot Plant for Fischer-Tropsch Synthesis by the US Method using Fluidized Bed of Iron Catalyst
- Z. Design of a Pilot Plant for Separation of the Fischer-Tropsch Synthesis Products
- aa. Report on Water-Gas Shift Reaction under Pressure using Fixed and Fluidized Beds
- bb. Planned Catalyst Production Plant at Severo-Donetsk
- cc. Possible Cyclamate Production Plant near Rubeshnaya
- dd. Phthalic Anhydride Production Plant at Rubeshnaya

a. Design of a Urea Plant

- (1) Mr. Harold, Gail, [] had been requested to design a Urea Plant of 5000 metric tons per month capacity. (Lacking experience with a plant of this size, [] submitted instead two proposals for pilot plants of 3.6 and 30 metric tons per month capacity, differing only in equipment size. In these designs, liquid ammonia and carbon dioxide were to be pumped through two reactors each consisting of series-connected, horizontal, steam-jacketed tubes, placed one above the other. These reactors were to be operated at 150-160°C and 130°C respectively, under a pressure of 100 atm, and with a throughput time of one hour in each reactor. The pressure was twice as high as had been used at Leuna, but was recommended because the reaction is favored by higher pressures. It was assumed that ammonium carbonate would be the main product from the first reactor, and that urea and water would be obtained from the second reactor. This product was then to be flashed in a column containing sieve plates from which the ammonia and carbon dioxide vapor would be recompressed for recycling, while the urea-water mixture would be drawn off for drying over a drum drier. It was suggested that monel, illium, high silicon steel, or lead-lined reactors be used.)
- (2) [] it was intended to locate this plant at Severo-Donetsk. The urea was probably to be used for urea-formaldehyde resins, such as Kaurit. [] never heard of the use of urea resins as anti-radar coatings for submarines.

b. Design of an Experimental Laboratory for Severo-Donetsk

- (1) Dr. Schmidt worked on this design. []

c. Report on Acetic Acid and Esterification

- (1) Dr. Genssner completed this report at Severo-Donetsk. It had been assigned to him at the Karpev Institute. It was to cover the Leuna work on acetic acid, and include a general review on esterification.
- (2) During the war, experiments were conducted at Leuna to produce acetic acid from methanol and carbon monoxide under a pressure of 700 atm. []

d. Construction and Installation Plans for Dismantled Leuna Equipment

- (1) Dr. Bode worked alone on this assignment. He was [] transferred to the Construction Office at Severo-Donetsk, where he had to examine construction plans which had been evacuated from Leuna, and work out new construction plans for the dismantled Leuna machinery and equipment at Severo-Donetsk. This work kept him busy until April 1950.

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- (2) He was the only one [] who was permitted to enter the storage enclosure, where the dismantled Leuna equipment was lying in the open. [] his drawings were designed exclusively for use in the Severo-Donetsk plant. He was much concerned with construction and installation plans for motors and engines. He was never permitted to enter the Severo-Donetsk plant proper.

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e. Questionnaire on Ammonium Nitrate

- (1) This collective assignment, Dr Bode excluded, was given to [] Mr. Nikitenko, and was accepted by him after its completion. It consisted of about 7-10 questions, such as the following:

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- (a) How can the danger of explosion be diminished?
- (b) How can the strewing quality of the product be improved?
- (c) How can caking be prevented?
- (d) What is the best method of producing crystals of a definite grain size?
- (e) What are the best conditions for storage and transportation?

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f. Memorandum on Kaurit Leim or K-Glue

- (1) [] production of Kaurit Leim, or K-Glue. This glue is a partially polymerized urea-formaldehyde condensation product, whose final polymerization occurs in situ, after application, thereby producing an irreversible glue. The final polymerization, or setting, can be inhibited for about one month by the addition of ammonium salts, but then the addition of accelerators is required before application.

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- (2) Kaurit Leim was not produced at Leuna until after the war. [] the production was about 100 metric tons per month.

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g. Report on Silica-Alumina Catalysts

- (1) [] comprehensive report on the Leuna wartime production of catalysts containing silica and alumina.

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- (a) Production of Basic Materials: Silica Gel and Active Alumina
- (b) Production of Catalysts from Basic Materials
- (c) Properties of these Catalysts
- (d) Applications of these Catalysts.

The first part was completed in December 1948. The second part was worked on and completed during March 1949. The third part was written during April 1950. The fourth part was never started.

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- (2) Nothing was included in these reports which has not already been mentioned in describing [] work at Leuna, December 1939 - October 1946, except for a few minor items, known from the literature, such as the additional use of oil cracking catalyst for esterification reactions.

h. Production of Formaldehyde

- (1) Drs Gemassmer, Schmidt, and Gaib worked on this report describing

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1. Production of Propionaldehyde

- (1) Early in January or February 1949 [] worked on a report concerning the production of propionaldehyde at Leuna, but lacked essential data for more than a meager report based on theoretical considerations. In January 1950, however, Soviet-collected Leuna data from the archives of GIAP in Moscow were made available [] 25X1
- (2) At Leuna, propionaldehyde was produced by oxidizing normal propyl alcohol with air over a silver catalyst at 520°C and atmospheric pressure. Because of the large heat of reaction, the propyl alcohol was diluted with water to the azeotropic composition, 70% n-propanol, 30% water. For further heat absorption, and in order to compensate for the mass action effect of the 30% water, an excess amount of air was used. Because of the sensitivity of the catalyst to carbon dioxide and to small traces of iron, the water was prepared from steam condensate purified with peat charcoal, and the air was treated by washing first with caustic to remove carbon dioxide, then with water to remove traces of caustic, and finally by filtering through glass wool. Special alloy steel was required for all piping and equipment leading up to and including the catalyst chamber. 25X1
- (3) The silver catalyst was composed of silver granules, two-five mm in diameter, having a definite crystal structure. The catalyst was prepared electrolytically in a ceramic tank containing, as electrolyte, an air-agitated water-solution of silver nitrate and nitric acid. Lumps of silver, suspended in a bag, comprised the anode. A small horizontal rail, equipped with a mechanical scraper, served as cathode. [] The granules which fell from the cathode were collected, removed, washed, put into a crucible equipped with a tube, and heated to red heat, 500-600°C, in an electric furnace. Methanol vapors were then introduced through the tube in the crucible, and after several minutes, the granules, thus activated, were cooled and stored for use. Reactivation of used catalyst was accomplished in the same manner, except that a 24-hour digestion with concentrated nitric acid, followed by a 24-hour digestion with concentrated ammonium hydroxide, was required to assure complete removal of all iron and copper before the crucible treatment. 25X1
- (4) The process for the production of propionaldehyde was as follows: The azeotropic solution of normal propanol and water was preheated, vaporized, and mixed, in a nozzle-type mixer, with the excess air, preheated to 80-100°C. The resulting blend was introduced to the reactor at such a velocity that the heat of reaction maintained the catalyst bed at 520°C. The catalyst bed, composed of three layers with the largest granules on the bottom, was spread on a bronze screen supported on a perforated sheet of Deutro-16 alloy, which is roughly 18-8 Cr-Ni alloy containing also 2% Si and 1% Mn. Because propionaldehyde is not stable at 500°C, the products from the catalyst chamber were immediately cooled in a bundle of vertical tubes located directly under the catalyst bed, and forming an integral part of the reactor unit.
- (5) Products from the reactor were led to a second cooler, then to caustic neutralizer to remove organic acids, and then to a distillation column in which an aldehyde-water fraction was

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separated from bottoms containing residual products, water and unreacted propanol. The aldehyde-water fraction was distilled in an aluminum column to produce 97-98% propionaldehyde as finished product. The bottoms were processed in a third column to recover n-propanol-water azeotrope, which could be recycled.

- (6) In starting up, the catalyst chamber cover was replaced by another containing a radiant heater. As soon as the catalyst was up to temperature, 550°C, the heating cover was replaced by the operating cover, and the preheated vapor mixture introduced. The process was controlled by visual observation of the red-hot catalyst through a peep glass in the operating cover.
- (7) At Leuna the production was intermittent and averaged about 30 metric tons per month of propionaldehyde, which was used only for the production of trimethylol ethane.

3. Production of Trimethylol Ethane

- (1) Dr Gemassner [] completed this report during the months of January and February 1950. [] started on it about a year earlier, but were limited to theoretical considerations until the Soviet-collected Leuna data [] arrived from Moscow in January 1950. The report described the very expensive process used at Leuna during the war to produce trimethylol ethane, or P3, which could be used as a substitute for glycerine in the manufacture of alkyl resins by reaction with dibasic acids or anhydrides. It could also be used as an antifreeze. [] it was also nitrated at Troisdorf, near Cologne, to produce an explosive used in the tropics, but it was rumored the explosive properties were not too good. The name P3 stood for the 5 penta carbons and the 3 methylol groups in the molecule. The process involved the reaction of one molecule of propionaldehyde with three molecules of formaldehyde and one molecule of water to produce trimethylol ethane and formic acid with dimethylol propionaldehyde as an intermediate.
- (2) In practice, about a two-ton batch was charged into a wooden vat equipped with a stirrer and a coil. The latter could be used for water cooling or for steam heating. Included with the charge was a definite amount of calcium hydroxide, perhaps about 2% by weight, which was introduced in the form of milk of lime, and served as a catalyst for the above reactions. For the first two hours, the charge was maintained at 20-25°C by the use of cooling water, since at temperatures above 30°C condensation reactions would occur in the presence of lime. Sulfuric acid was then added to neutralize the lime, the cooling water was replaced by steam, and the charge was heated to 100°C over the course of one hour. After cooling, the calcium sulfate and any calcium formate which had settled out were removed by filtering through a filter press. The filtrate, containing P3, water, some calcium formate, and organic residue, was pumped into two tall towers, each of about 50 cubic meters capacity, where further settling of calcium formate could occur. Filtrate from the upper layers was pumped to the three-stage, countercurrent extracting units. [] three stages would be the optimum number, and were gratified to find, when the Leuna data arrived, that three stages had proven best in actual production.

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- (3) In the old process, isobutylene, composed of one part isobutanol and two parts diisobutylketone, was used as the extractor. It was not very selective, and was saturated by about 1% of P3. In the last months of the war it was found that isobutanol alone was a better extractor, both in regard to selectivity and solubility of P3, which was about 5%. This mono alcohol extractor was called Monol. Each of the three-stage, countercurrent extracting units had a capacity of about six cubic meters. Filtrate occupied about three-fourths of the volume of each. Bottoms were eventually discarded or else hydrogenated for fuel. The extract was stripped of its water-soluble P3 content by passage through two water towers. The stripped extract was distilled before reuse in order to remove organic residues which could be discarded or hydrogenated for fuel. The water solution of P3 was concentrated in a steam jacketed kettle equipped with several fractionating plates. The concentrated P3 melt was dried in a vacuum drum drier. The final P3 product was a colorless crystal which was stored and shipped in drums. War production of P3 at Leuna was about 50-60 metric tons per month.

k. Production of Glycerine

- (1) This report by Drs Herold and Schmidt was supposed to cover the method used at Heydebreck, but no data whatsoever were available; therefore, Drs Herold and Schmidt wrote a very short theoretical report based on US literature.

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l. Production of Oppanol C

- (1) Dr Gemassmer [] worked on this memorandum. Since Oppanol C was a plastic development at Oppau, and had not been produced at Leuna, [] forced to rely almost entirely on data Gemassmer had in his private notes.

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- (2) Oppanol C is made by polymerization of isobutylene at -80°C using boron trifluoride as a catalyst. The K-wert, or condensation value, is about 1000 monomers, and is controlled by the addition of small amounts of diisobutylene, less than 0.002% being sufficient to restrain the chain growth to the desired length. In practice, the isobutylene is dissolved in one stream of liquid ethylene (bp -103°C), and the boron trifluoride and diisobutylene dissolved in a second stream of liquid ethylene. These streams are mixed by pouring them together on a horizontal, stainless steel, endless conveyor belt where the isobutylene is polymerized and the ethylene solvent vaporized. Ethylene vapors are recovered, compressed, condensed and reused. The product is a dry, brittle film, which breaks into flakes as it leaves the conveyor belt.

- (3) Oppanol C is thermoplastic, highly elastic, and can be mixed and compounded with natural rubber. It makes excellent elastic sealing rings.

m. Production of Mersol

- (1) Drs Herold, Geib, and Schmidt divided the work on this report as follows: Dr Herold submitted a general review; Dr Geib worked on, but never completed, a theoretical review on the sulfochlorination of hydrocarbons; and Dr Schmidt completed a review on the separation of reaction products by extraction.

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- (2) Mersol is produced by reacting straight chain hydrocarbons, containing 12-16 carbon atoms with sulfur dioxide and chlorine vapor at normal temperatures and pressures in quartz reactors under the influence of ultraviolet light. By neutralizing with caustic soda, Mersol forms water-soluble sulfonates, known as Mersolate, which have good detergent properties.
- (3) Mersol was produced at Leuna during the war. Dr Spohn is the present chief of the Mersol plant at Leuna. [redacted]

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a. Production of Fatty Acids by Alkali Fusion

- (1) [redacted] report [redacted] described the method used at Leuna, during the war, to produce fatty acids by alkali fusion. The process was as follows: higher straight chain or alpha methyl alcohols containing six-eight carbon atoms were reacted with caustic soda in an autoclave at 240-280°C and 10-15 atm to produce sodium salts of the corresponding acids and free hydrogen. The sodium salts were treated with dilute sulfuric acid to liberate the organic acids which were then removed by vacuum distillation.
- (2) The war production was about 10-20 metric tons per month of higher fatty acids. This product is no longer being made at Leuna.

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b. Production of Adipic Acid

- (1) This report was [redacted] to describe the wartime production of adipic acid at Leuna. (It was prepared by oxidizing cyclohexanol with 67% nitric acid at 50-60°C and atmospheric pressure in a stainless steel vessel accompanied by vigorous stirring to avoid detonation of an explosive intermediate assumed to be the nitric acid ester of cyclohexanol. Vapors arising from the reaction mixture were absorbed in milk of lime to produce a fertilizer containing calcium nitrate and calcium nitrite. The reaction mixture was fractionally crystallized, centrifuged, the crystals redissolved and recrystallized and after separation dried in a drum drier using heated air. The main by-products were oxalic and succinic acids.) A portion of the mother liquor was recycled.
- (2) (This plant was dismantled by the Soviets and probably sent to Dzerzhinsk together with the hexamethylenediamine plant equipment. During the war years 1943-44, Leuna produced in Bldg No 478 about 30 metric tons per month of hexamethylenediamine. [redacted] these plants both went to Dzerzhinsk because hexamethylenediamine and adipic acid are the raw materials which are copolymerized and woven to produce nylon, and the Soviet, Ivan Ivanovich, who had been at Leuna and was very much interested in perlon production, also went to Dzerzhinsk.) Furthermore, Drs Striegler and Meier, who were sent to Dzerzhinsk in October 1946, reportedly had a pilot plant there for the production of caprolactam, which is polymerized to give the product from which perlon fabric is woven.

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c. Report on Gxe Process

- (1) Dr Gemassner submitted this report on the Gxe process, which is the production of aldehydes from Fischer-Tropsch olefins by treatment with carbon monoxide and hydrogen. The aldehydes can then be reduced in a second step to give the corresponding alcohols.

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- (2) Prof Roelen worked on this process for Ruhr Chemie before the war. The Ruhr Chemie gave its results on Fischer-Tropsch synthesis to I. G. Farben, and the two developed the Oxo process together. Both I.G. Ludwigshafen and I.G. Leuna worked on it. At Leuna, the raw liquid olefin was mixed with cobalt catalyst to form a slurry which was pumped into the reactor at 200 atm. Carbon monoxide and hydrogen were recycled through the reactor, and the liquid reaction mixture was drawn off and filtered to remove catalyst. The aldehyde fraction was then reduced with fresh hydrogen in a second reactor at 200 atm over a fixed bed of Fischer-Tropsch catalyst consisting of cobalt, magnesia, and thoria on kieselguhr.
- (3) Since carbon monoxide was poisonous to this catalyst, the small amounts entering in the crude aldehyde feed were stripped and removed by cycling the hydrogen through the reacting mixture in the reduction vessel and then through a methanization oven where the carbon monoxide was converted into methane. This oven operated at 180-200°C and 200 atm, and contained the same black oxide catalyst as was used in ammonia synthesis. The reduced alcohols of 8-16 carbon atoms, were drawn off from the bottom of the reduction vessel and separated by fractional distillation. They were desired especially for the production of synthetic detergents.

q. Report on Synol Process

- (1) [redacted] this report which was prepared by Dr Genassmer contained only material presently available in the literature. 25X1

r. Design of Tunnel-Type Drying Oven

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- (1) Dr Schmidt worked on this design. [redacted]

s. Questionnaire on Brown Oxide Catalyst

- (1) This questionnaire [redacted] concerned the production of brown oxide catalyst at Heydebreck, Silesia. This plant had been built during the war, primarily for the production of isobutanol, which by catalytic dehydration could be converted to isobutylene, which could then be polymerized and catalytically hydrogenated to isooctane for use in aviation fuels. The plant at Heydebreck produced about 100,000 metric tons per year of isobutanol. 25X1
- (2) There were about fifteen questions in all, such as the following: conditions of precipitation, production temperatures, production of a chromium nitrate solution, technological questions on the construction of a screw conveyor, questions concerning disposal of waste waters containing iron and chromium, etc.

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t. Report on Production of Ammonia Catalyst

- (1) [redacted] the first part of this report [redacted] described the method of producing ammonia catalyst at Leuna. This catalyst is a black oxide catalyst containing, mainly, ferric oxide with 2% potassium oxide. 25X1

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- (2) ☐ the second part ☐ discussed the possible use of this catalyst in the following syntheses:

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- (a) The KWSy or hydrocarbon synthesis for producing saturated hydrocarbons directly from carbon monoxide and hydrogen.
- (b) The Michael process for the production of olefins from carbon monoxide and hydrogen by recycling the gaseous products.
- (c) The Buftschmidt process for the production of olefins from carbon monoxide and hydrogen by recycling not only the gaseous products but also the cooling oil in which the fixed catalyst bed is immersed, and in which the olefins are dissolved.
- (d) The Synol process for producing oxygenated hydrocarbons directly from carbon monoxide and hydrogen, instead of from olefins as in the Oxo process.
- (e) The production of hexamethylenediamine by hydrogenation of adipic dinitrile. Although the laboratory catalyst was Raney nickel, black oxide catalyst was used in the plant.

u. Memorandum on Hydrogen Purification with Alkaline Copper Solution

- (1) Dr Herold wrote this memorandum on the Leuna hydrogen purification plant

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- (2) Dr Herold later remarked that he had unintentionally reported a carbon monoxide content of 0.001%, instead of 0.01%, in the hydrogen gas after purification with alkaline copper solution.

v. Production of Higher Alcohols by Aldehyde Reduction

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- (2) It is only in the case of the Oxo Process, where the aldehydes cannot be readily separated, that they are reduced to produce alcohols of 8-16 carbon atoms. Also in higher alcohol fractions, aldehyde impurities may be reduced to corresponding alcohols to facilitate separation in subsequent fractional distillation.

w. Production of Methanol and Isobutyl Oil

- (1) This report by Dr Herold included a flow sheet and description of the Synol process used to produce oxygenated hydrocarbons, primarily, methanol and a mixture of other alcohols comprising isobutyl oil. In this process, a mixture of carbon monoxide and hydrogen gas are passed at 350-400°C and 220-230 atm over a methanol catalyst internally cooled with cold synthesis gas. The catalyst contains about 33% zinc oxide, 66% chromic oxide, and one-two per cent potassium oxide.

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x. Separation of Alcohols in Isobutyl Oil

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- (1) Dr Gemassner described in this report how, by hydrogenation of aldehyde impurities and by fractional distillation, the alcohols in isobutyl oil could be separated.

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7. Design of a Pilot Plant for Fischer-Tropsch Synthesis by the US Method using Fluidized Bed of Iron Catalyst

- (1) Mr. Makarov, who had been Deputy General Manager of Leuna until January 1950, arrived at Severo-Donetsk in April 1950, and gave [] 25X1 entire group, including Bode, the above assignment. He also requested the design of a pilot plant for separation of the Fischer-Tropsch synthesis products.
- (2) [] did not have a laboratory to work in but were well supplied with US literature which we received partly in the original and partly in photocopies sent directly from Moscow. [] also [] available, in Brennstoffchemie, a 1949 article by Mr. Fiehler, friend of Mr. Tropsch, describing the American Fischer-Tropsch synthesis [] which [] claimed that by recycling, a conversion of 90% was possible. 25X1
- (3) Although the normal American operating pressure was 15-20 atm, [] designed a pilot plant in which, for experimental purposes, pressures could be varied up to 50 atm, and temperatures from 400°C down to 180°C. The pilot plant could thus be used at the higher temperatures to produce hydrocarbons, as in the American process, or at the lower temperatures to produce oxygenated hydrocarbons, as in the German Synol process. [] assumed the higher temperatures would be necessary in the production of straight hydrocarbons in order to prevent the occlusion of higher paraffins on the fluidized catalyst. The plant was designed to treat 180-200 cubic meters of synthesis gas per hour, containing two parts of hydrogen per part of carbon monoxide. It was assumed that the synthesis gas would be available at the plant and that a conversion of 50% would be obtained without recycling. 25X1
- (4) The feed gas was to be electrically preheated before entering the reactor at a linear velocity of about 1 ft per sec under a pressure of 15-25 atm. The gases from the reactor were to be separated from the fluidized catalyst in a cyclone separation. Catalyst was to be recycled for about one hour before being replaced. The hot gases were to be filtered through a ceramic hot filter. The liquid waxes, high boiling paraffins, and catalyst fines were to be discarded. The filtered gases were then to be passed through a water-cooled heat exchanger, from which the higher boiling paraffins and organic acids would be separated and treated with sodium hydroxide solution to remove the organic acids. For an actual plant, the acids would be removed by other means.
- (5) Remaining gases would be cooled in a liquid ammonia cooler to no less than about 0°C to prevent icing in the liquid hydrocarbon fraction. The methane, ethane, and ethylene in the residual gases would be removed by absorption in oil or charcoal. Unreacted synthesis gas would be metered and burned as fuel, or recompressed and recycled.
- (6) The plans included six-eight tanks, each of one-two cubic meters capacity, in which to store the liquid hydrocarbons.)
- (7) Mr. Makarov intended to build this pilot plant as soon as an experimental laboratory was available.

8. Design of a Pilot Plant for Separation of the Fischer-Tropsch Synthesis Products

- (1) The Fischer-Tropsch synthesis products could be divided into the water layer containing low alcohols, ketones, etc., and the oily

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layer containing olefins and higher oxygenated products. Each layer required special treatment. The first step with each layer was to remove the three-four carbon atom fractions in topping stills. These fractions could then be polymerized or alkylated to isooctane and the like.)

- (2) Several methods were proposed for treating the residual water layer. Among them were:
- (a) Simple fractional distillation to separate the products
 - (b) A combination of extraction and fractional distillation of both extract and residue
 - (c) A combination of esterification and fractional distillation
 - (d) Mild reduction of aldehydes and ketones to corresponding alcohols followed by fractionation or esterification and fractional distillation
 - (e) Dr Herold suggested oxygenation of aldehydes and ketones to produce organic acids
- (3) The methods proposed for treating the residual oily layer included:
- (a) Simple fractional distillation to separate the products
 - (b) Catalytic reforming over active clay to increase the octane rating of the gasoline produced. The active clay would also serve as a dehydration agent to yield additional olefins for the reforming process
 - (c) Mild reduction of aldehydes and ketones to corresponding alcohols, followed by esterification with boric acid or boric anhydride. The hydrocarbons could then be separated from the boric acid esters, and the esters, because of their wide range of boiling points, could then be readily separated by fractional distillation. The alcohols could be regenerated by treating the separated esters with sulfuric acid. This method was originally an analytical procedure, but it had been worked out at Leuna during the war for the plant production of alcohols from the Synol process
 - (d) Same as (c), but using a higher dibasic fatty acid, such as adipic acid, instead of boric acid.

The catalyst specified for the mild reduction of aldehydes and ketones in both the water layer and the oily layer was the Leuna catalyst No 1930 composed of 1.25 mol Cu, 0.50 mol Cr_2O_3 , and 1.00 mol ZnO.

- (4) The pilot plant design included some calculations of distillation columns, extractors, reactors, oxidizers, and reaction vessels for esterification. The consumption of synthesis gas was to be 160 - 200 cbm per hour. The final design was completed in December 1950, and turned over to Mr Makarov in two books of more than 200 typewritten pages each.)
- (aa) Report on Water-Gas Shift-Reaction under Pressure using Fixed and Fluidized Beds
- (1) The entire group was given this assignment in January 1951. As reference we had a Soviet report by Major Murzin, on the unpressurized

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Leuna method. This report had been written partly by Mursin and partly by Baumann, who is chief of the conversion plant at Leuna. Major Mursin is presently chief of a branch of the GIAP at Severo-Donetsk. [redacted] had little material on the conversion under pressure, except for an issue of the US publication "Industrial and Engineering Chemistry", 1949 or early 1950. [redacted]

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- (2) When steam is passed over glowing carbon or coke, an equimolar mixture of carbon monoxide and hydrogen, known as water-gas, is formed. Additional steam will react with this water-gas, in the presence of brown oxide catalyst, to form carbon dioxide and hydrogen, the so-called water-gas shift-reaction. By removal of the carbon dioxide, hydrogen is available for ammonia synthesis.
- (3) In early practice, the water-gas from the generators was first purified to remove the sulfur by a two-stage process in which first alkazid or diethylol amine was used to remove hydrogen sulfide and carbon dioxide, and then active charcoal was used to remove residual organic sulfur. The charcoal could be regenerated by extraction with ammonia solution to form ammonium polysulfide. Sulfur from this compound was sent to Bitterfeld for the production of sulfuric acid.
- (4) The purified water-gas was then washed with water at 70-80°C in a wooden grid column from which it left, at atmospheric pressure, containing about 50% water vapor. It then passed through a steam injector which provided excess steam and the pressure necessary for flow through a heat exchanger and water-gas shift reactor at 360°C, and due to the exothermic heat of reaction left at about 460°C. It was partially cooled in the heat exchanger already referred to and entered a second wooden grid column where it was further cooled with water spray at 67-70°C. It was then compressed to 20 atm, purified from carbon dioxide by water washing, and further compressed to 200-250 atm before being piped to the hydrogen purification plant. The water spray, heated to 70-80°C in the process, was circulated to the first wooden grid column where it was cooled to 67-70°C and recycled, with the addition of make-up water.
- (5) The brown oxide catalyst contained about 90% ferric oxide, 6% chromic sesquioxide, and 4% water and other impurities. Although it was sulfur resistant, it was necessary to remove sulfur from the water-gas since the black oxide catalyst in the ammonia synthesis was poisoned by the presence of sulfur in the hydrogen.
- (6) The primary cost in the process described was the cost of the injector steam and the cost of compression following the water-gas shift-reaction. Since this reaction produced two molecules of hydrogen plus one of carbon dioxide from one molecule of hydrogen plus one of carbon monoxide in the water-gas, it followed that compression costs could be reduced one-third by compressing the water-gas to 20 atm and conducting the shift reaction under that pressure. Cost of injector steam could be reduced by taking advantage of the exothermic heat of reaction to generate steam from a water spray within the shift reactor. This procedure would have the further advantage of providing a more uniform and lower reaction temperature which favored the equilibrium. Excess water vapor was used to displace the equilibrium to the right. [redacted] the equilibrium was uninfluenced by the higher pressure.
- (7) The reactor proposed had a capacity of 10,000 cubic meters per hour of fresh water-gas. It was about 2.6 meters in diameter and nine meters high. The upper and lower portions each contained about five

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cubic meters of catalyst of 1.13-1.14 bulk density. The center portion, over which water was sprayed, consisted of a six-ten cm layer of Raschig rings.

- (8) The Heydebreck pressure reactor had been similar in design, but higher and with several water spray levels.

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(bb) Planned Catalyst Production Plant at Severe-Donetsk

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- (1) [redacted] at Severe-Donetsk [redacted] indications that the plant might be extended to include a large catalyst production plant.
- (2) In July 1950 Severe-Donetsk was incorporated into the GIAP, the Government Institute for Nitrogen Production. At that time, Mr Mursin talked [redacted] about plans for a large catalyst plant. These plans had been worked out at Soviet request by the KIB, the Construction Engineers Bureau, which used to be part of Leuna but was entirely separated from it in organization after the war when Leuna was a member of the SAG, Soviet Inc, while the KIB was "people owned". Although these plans, which were contained in seven volumes in the Severe-Donetsk archives, were preliminary in the sense that the location of the plant and the source of water and power supply were not identified, they were, nonetheless, executed in great detail. They called for the following production:

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- (a) Brown oxide catalyst for water-gas shift-reaction; 500 metric tons per month. This is about six times larger than present requirements at Leuna.
 - (b) Black oxide catalyst for ammonia synthesis; 100 metric tons per month.
 - (c) Cobalt, magnesium oxide, thorium oxide, on kieselguhr carrier for Fischer-Tropsch syntheses; 200 metric tons per month.
 - (d) Zinc oxide, chromium oxide, potassium oxide catalyst for methanol-isobutyl oil synthesis; 30-40 metric tons per month.
- (3) Other catalysts for [redacted] planned production capacity were:
- (a) Alumina, kaolin catalyst for methyl amine synthesis.
 - (b) Nickel sulfide or tungsten sulfide catalysts for hydrogenation. The small amount of hydrogen sulfide in the hydrogen feed gas keeps the catalyst active.
 - (c) Copper, chromium oxide, zinc oxide catalyst, called Leuna No 1930, for after-hydrogenation in the synthesis of higher alcohols.
 - (d) Zinc oxide, pumice catalyst, and F-coal, which is an active coal for the desulfurization of synthesis gas.

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(cc) Possible Cyclonite Production Plant near Rubeshnaya

- (1) There is a plant between Severo-Donetsk and Rubeshnaya, but nearer the latter, on its southern periphery, where cyclonite is supposedly being produced. Cyclonite is trinitrotrimethylesternamine and is also known as hexogene and RMX.
- (2) In July 1948 this plant was still not in operation, and German prisoners of war were working in the ruins. By about June 1950, however, a single smoke stack was operating intermittently, as if the plant were starting up. This plant receives its power from Proletarsk, near Severo-Donetsk.

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(dd) Phthalic Anhydride Production Plant at Rubeshnaya

- (1) The Soviets are making phthalic anhydride at Rubeshnaya, which is about 14 km from Severo-Donetsk. The production process is by oxidation of naphthalene over a fixed bed of V_2O_5 catalyst.
- (2) Dr Werner Keller, who is one of the scientists deported from the chemical plant at Wolfen to Rubeshnaya, the Soviets requested that Dr Schuster (fnu), another Wolfen scientist, modify this method of production from fixed bed to fluidized bed. The vanadium oxide catalyst would be drawn off continuously, regenerated, and returned to the reactor, thereby making the oxidation a continuous process. Dr Schuster worked on this project in the laboratory at Rubeshnaya and finished it, making a pilot plant which could be used for small production.

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